

06915USA

TITLE OF THE INVENTION:

COATING MATERIAL

DESCRIPTION FIELD OF THE INVENTION

[0001] The present invention relates to conductive nanoparticles with improved properties and transparent coatings made of the same.

5

BACKGROUND OF THE INVENTION

[0002] Transparent conductive coatings are of great significance and are well known among other things for displays (e.g., CRT, LCD, and OLED, ...) and/or antistatic coatings. Standard methods for manufacturing comprise among others first the gas phase coating (e.g., sputtering, CVD, PVC, ...) of flat glass with thin, conductive coatings that are transparent in the visible world. Along with (precious) metals (e.g., precious metals), conducting or semi-conducting doped oxides such as ATO (SnO₂:Sb), FTO (SnO₂:Sb), FTO (SnO₂F), AZO (ZnO:A1) or ITO (In₂O₃:Sn) are used as coating materials. As a rule thick coatings are obtained via these methods. This is known as the standard method for the coating of flat glass in order to generate high-quality coatings for flat glass, whereby a fully developed technology is available.

[0003] The sputter facilities that are used for glass coating are quite expensive (2 to 3-figure million amounts) and work profitably only in the case of very great large operational capacity (coating of several 100 T m²/a). In addition, from a technical standpoint they are very expensive and have a very such facilities require high material consumption; because when either the material that is to be deposited vaporized or a target is are vaporized, steam the vaporized material is only partially deposited on the substrate to be coated, while the rest settles somewhere in the interior of the machine. In addition to that Further, such facilities are inflexible, so such that small or special runs are hardly feasible, in particular impractical since almost exclusively only flat geometries can be coated using sputter facilities; Other geometries are only possible to a limited extent and the corresponding facility must be rebuilt with each change of geometry. This is somewhat of a problem for automobile glazing, because there are no absolutely flat

06915USA

automobile panes. ~~even the attempt to sputter~~ Sputtering flat panes and then bending them has not succeeded up to this point. Even coating of polymers and films is only possible to a limited extent.

~~[0003][0004]~~ There have also been approaches for realizing such coatings via ~~the use of using~~ conductive nanoparticles (e.g. ITO), ~~among others, s~~ Such methods exhibit as advantages as a simple coating technique, (e.g. via wet-chemical methods (such as painting, spraying, pressing, dipping, and spin-coating, ...), thereby permitting: making possible (i) the direct application of coatings onto structures, have (ii) a low technical expense with correspondingly low investment costs as a consequence, (iii) are geometry independent, (iv) make better use of the material, (v) have greater flexibility, and (vi) permit a coating of polymers and films.

~~[0004][0005]~~ However, ~~e~~ One basic requirement is the availability of, for example, e.g. IT nanopowder suitable particle size and the redispersibility with the corresponding properties. It is known from U.S. Patent- ~~Number-~~ 5,518,810 (Mitsubishi) that a specific shade of color correlates with optimum properties ~~(e.g., with reference to the infrared shielding).~~ Typically blue is an indication of a high number of oxygen defects, thus a high charge carrier density, which is caused by oxygen defects. This is, as a rule, generated in ITO by annealing the powder or coatings of the powder under inert gas or reducing atmosphere. Specifically, to be precise in the case of temperatures above 250° C. ~~This process results in blue powder having a higher conductivity than yellow powder that has not been retreated under reducing atmosphere, and this process results in coatings that have been compressed-reduced (e.g., by heat treatment at 500° C. in the air after inert gas/reducing treatment at temperatures above 250° C. show significantly higher conductivities).~~ A subsequent temperature treatment of ITO coatings under reducing/inert atmosphere at temperatures above 250° C. is state of the art. However, with many technical applications such a subsequent treatment is often not desirable or not possible, since the coated objects are destroyed at the required temperature, (e.g., with CRT or conductive and/or antistatic coatings on plastic). At the same time, however, the requirements and the need for highly conductive, transparent coatings on plastic are also increasing.

~~[0005][0006]~~ The object of the present invention ~~consists in relates to~~ making something new available for industrial application.

~~[0006][0007]~~ The solution of this task will be claimed in independent form.

06915USA

BRIEF DESCRIPTION OF THE DRAWINGS

- [0008] Figure 1 is a drawing of the apparatus used for making conductivity measurements.
- 5 [0009] Figure 2 is a graph showing the transmission of the inventive powders.
- [0010] Figure 3 is a graph showing the conductivity of the inventive powders.

DETAILED DESCRIPTION

- 10 ~~[0007]~~[0011] Consequently, in accordance with the invention an improvement results in the conductivity of coatings which contain ITO nanoparticles, ~~to be precise with a~~ simultaneous higher transparency in the visible world with a simple coating technique. Examples of such techniques comprise, in particular by means of wet-chemical methods (e.g., painting, spraying, pressing, dipping, and spin-coating) among other techniques with lower technical expenses and lower investment costs.
- 15 ~~[0008]~~[0012] ~~In the process of~~ The better conductivity obtained by the inventive process opens up new fields of application and allows less material (ITO) to be employed for the same conductivity, which leads to more favorable end products. Typically it should be assumed that, when for example coatings are produced, which consist of conductive starting powders and/or in which conductive particles are integrated in a matrix, the
- 20 conductivity of these coatings will be higher, the higher the conductivity of the starting powders or particles is. However, the invention has recognized that coatings with better conductivity can be produced from yellow powder with actually poorer conductivity than can be produced from blue powder, which as such exhibits a higher conductivity. This will be ~~verified with the help of some typical examples~~ illustrated by the following
- 25 examples.

EXAMPLES

EXAMPLE 1

- ~~[0009]~~[0013] Yellow ITO Powder is Produced in the Following Manner:
- 30 ~~[0010]~~[0014] Nanocrystalline $\text{In}_2\text{O}_3/\text{SnO}_2$ (ITO) powders are produced from an aqueous solution by means of a coprecipitation process in which soluble In or Sn components are

06915USA

precipitated by pH value increase. In this example the concentrations of these compounds were selected in such a way that the Sn concentration amounted to 5 at. % relative to In. However, in principle the Sn concentration can be arbitrarily adjusted.

- After separation of the reaction product it is dried and annealed at 300° C. under normal atmosphere for 1 hour for the purpose of setting the crystalline phase. Then the crystalline $\text{In}_2\text{O}_3/\text{SnO}_2$ is divided into different samples and the individual samples are after-annealed for different periods of time under forming gas at 300 C. (see Table 1).

- ~~[0014]~~[0015] Powders with color indexes that range between the maximum values listed in Table 1 (IT-05 HCB and IT-05 HCG) as well as coatings with yellow indexes between the corresponding values from Table 1 can also be obtained by mixtures of the various powders.

TABLE 1

Sample Name	Duration of treatment	Color index	Yellow index
IT-05 HCB	1.5 hour	X = 0.294, y = 0.332	0.12
BG9010	1 hour	X = 0.301, y = 0.341	1.17
BG8515	50 min	X = 0.306, y = 0.346	2.97
BG8020	45 min	X = 0.310, y = 0.351	4.63
BG7030	40 min	X = 0.318, y = 0.361	6.4
BG5050	30 min	X = 0.338, y = 0.381	12.66
IT-05 GN	1 hour, N ₂	X = 0.404, y = 0.418	22.7
IT-05 HCG	--	X = 0.414, y = 0.421	23.8

- ~~[0012]~~[0016] Out of yellow ITO powders of to a large extent practically identical particle sizes and with identical dispersion coatings containing yellow ITO powder were prepared, separated with an organic binder by means of spin-coating, and the coatings were dried at 120° C. for 1 hours. The resulting coating thickness and obtained transparent composite coatings amounted to 3 µm. The conductivity of the coating with blue powder amounted to 1.8 kOhm/square; the conductivity of the coating of yellow powder was 0.8 kOhm/square. The yellow index of both coatings was measured with a color pen (Dr. Lange), and evaluated in accordance with DIN 6167 and ASTM D 1925

06915USA

(standard illuminant C; standard viewer 2°). While the yellow index of the coating of blue powder was below 0.1, the yellow index of the coating made of yellow powder was 23.8.

EXAMPLE 2

5 ~~[0013][0017] Out of yellow ITO powders and of blue ITO powders of to a large extent practically identical particle sizes and with identical dispersion~~ Transparent coatings containing yellow ITO powder and blue ITO powder were prepared separated by means of spin-coating on the front plate of a kinescope (CRT), ~~to be precise with a~~ The coating had thickness of 100 nm. After a brief period of drying at room temperature, in order to protect the ITO coating from scratching it was post-coated with a solution of a hydrolysable Si alcoholate by means of spin-coating. The double-coated substrate was heated for 30 minutes at 180° C. After cooling off, the conductivity of the two-coat system is 8.5 k Ω for the blue powder and 6.1 Ω for the yellow powder. The surface resistances of the coatings were determined using a 4 point conductivity meter (Loresta GP, Mitsubishi Chemical Corporation).

~~[0014]~~ In the measurements the conductivity of the powders was determined with the arrangement of measuring instruments outlined in FIG. 1. Referring now to Figure 1,

~~[0015][0018]~~ This arrangement of measuring instruments for determination of the conductivity of the powders shows a measuring instrument (multimeter) with reference number 1, a glass pipe with reference number 2, powder with reference number 3, with reference number 4 an extrusion press ram made of steel and with 5 indicates applied pressure.

~~[0016][0019]~~ The measuring instrument described in FIG. 1 was filled with 3.3 g each of powder. Then the upper ram was introduced and contacted with the measuring instrument. The upper ram was then weighted with the various weights and the resulting resistance was measured via the compressed powder charge using a commercial multimeter. The measurements resulted in the following readings:

Weight (kg)	Pressure (kPa)	Resistance (Ohm)	
		Blue powder	Yellow powder
1	77	42	1140
1.5	115	32	820
1.8	138	28	640

06915USA

[0017][0020] The color values of the powders were as follows:

[0018][0021] Blue: $x=0.294$; $Y=0.332$

[0019][0022] Yellow: $x=0.414$; $Y=0.421$

- 5 [0020][0023] The characterization of the powders takes place via conductivity and shade of color, the characterization of the coatings takes place via yellow index and conductivity.

06915USA

CLAIMS

1. Conductive, infrared-absorbing coating material consisting of indium tin oxide, characterized by the fact that the yellow index is above 15.

5 2. Coating made of a conductive powder in accordance with claim 1.

10

06915USA-TRACK.DOC

06915USA

ABSTRACT OF THE DISCLOSURE

The invention relates to a conductive infrared-absorbing coating material consisting of indium tin oxide. According to the invention, said coating material has a
5 yellow index of above 15.

10

06915USA-TRACK.DOC